(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
17 June 2004 (17.06.2004)

PCT

(10) International Publication Number
WO 2004/051227 A1

(51) International Patent Classification[7]: G01N 1/00, 1/34

(21) International Application Number:
PCT/GB2003/005242

(22) International Filing Date: 1 December 2003 (01.12.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0228006.3 30 November 2002 (30.11.2002) GB

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(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(regional)*: ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2004/051227 A1

(54) Title: AUTOMATIC THERMAL DESORPTION APPARATUS AND METHOD

(57) Abstract: An analytical apparatus for automatically carrying out a plurality of analytical steps, which apparatus includes: a releasing device for releasing a sample from a sampling tube; a device for analysing a first portion of the released sample; a collecting device for collecting a second portion of the released sample; a device for re-releasing the collected said second portion of the sample; and a device for analysing the re-released portion of the sample.

AUTOMATIC THERMAL DESORPTION APPARATUS AND METHOD

The present invention relates to an analytical apparatus and, more particularly, to an automatic thermal desorption apparatus, and a method for carrying out an automated analytical procedure.

It is known to provide a thermal desorption apparatus comprising an autosampler for automatically selecting one of a plurality of sampling tubes stored therein, heating the tube to desorb a sample from a sorbent material contained in the tube and then providing a flow of inert gas through the tube to drive the sample therefrom.

Known apparatus further comprise means for analysing a first portion of the sample driven from each sampling tube and for collecting a second portion of the sample in a respective collecting tube, for subsequent analysis.

However, a drawback of such known apparatus is that, following the analysis of samples released from a number of sampling tubes, their respective collecting tubes must be manually loaded into the autosampler for a subsequent analysis to be carried out.

A further drawback of such known apparatus is that automatic storage means must be provided for storing the collecting tubes in which respective portions of the samples released from each of the sampling tubes are collected, independent of the autosampler in which the sampling tubes are stored.

We have now devised an arrangement which overcomes the limitations of known analytical apparatus.

In accordance with the present invention, there is provided an

analytical apparatus for automatically carrying out analytical steps, which apparatus comprises:
device for releasing a sample from a sampling tube;
device for analysing a first portion of the released sample;
device for collecting a second portion of the released sample;
device for re-releasing the collected said second portion of the sample; and
device for analysing the re-released portion of the sample.

The apparatus may be arranged to select the sampling tube from a plurality of tubes stored in an autosampler. The apparatus thus obviates the requirement for the manual loading of collecting tubes into an autosampler, for a two stage analysis to be carried out on samples contained in a plurality of sampling tubes.

Preferably the apparatus is arranged to provide a comparison of the results from each of the two analysis stages.

Preferably, the second portion of the sample released from each sampling tube is collected either in the sampling tube itself or in a separate collecting tube or trap.

In the latter case, preferably either a single collecting tube or trap is used to collect, in turn, the second portion of the sample released from each of a plurality of sampling tubes, or respective collecting tubes are used to collect the second portion of each of the released samples, each of said respective collecting tubes preferably being selected automatically from a plurality of tubes stored either in the same autosampler as the sampling tubes or in a further autosampler.

Preferably, only a portion of the re-released sample is analysed, a second portion of the re-released sample being re-

collected, either in the sampling tube or in a further respective re-collecting tube, each of said respective re-collecting tubes preferably being selected automatically from a plurality of tubes stored either in the same autosampler as the sampling tubes and/or collecting tubes or in a further autosampler.

The apparatus may be arranged such that the sample released from the sampling tube is buffered, by collecting the sample in an intermediate tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the second portion of the released sample. Alternatively, the second portion of the sample released from the sampling tube, or subsequently collected and re-released, may be buffered, by collecting the sample in an intermediate tube or trap, prior to its collection/re-collection.

Also in accordance with the present invention, there is provided an analytical apparatus, comprising means for automatically carrying out, for each of a plurality of sampling tubes stored in an autosampler, the steps of:
selecting a sampling tube from said plurality of tubes;
releasing a sample from the sampling tube;
analysing a first portion of the released sample;
collecting a second portion of the released sample, either in the sampling tube or in a collecting tube selected from said plurality of tubes, with which said first tube is replaced in the autosampler.

The apparatus thus obviates the requirement for automatic storage means for storing the collecting tubes in which respective portions of the samples released from each of the sampling tubes are collected, independent of the autosampler in which the sampling tubes are stored.

The apparatus may also comprise means for automatically carrying out the further steps of releasing the sample collected in either the sampling tube or the collecting tube and analysing the released sample.

The apparatus may be arranged to analyse only a first portion of the sample released by said collecting means, a second portion of the released sample being re-collected, either in the sampling tube, the collecting tube or in a re-collecting tube selected from said plurality of tubes, with which either the sampling tube or the collecting tube is replaced in the autosampler.

The apparatus may be arranged such that, for each of the plurality of sampling tubes, the sample released from the sampling tube is buffered, by collecting the sample in a tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the second portion of the released sample. Alternatively, the second portion of the sample released from the sampling tube or by the collecting means may be buffered, by collecting the sample in a tube or trap, prior to its collection/re-collection.

According to a further aspect of the present invention, there is provided an analytical method, which method includes:
releasing a sample from a sample tube;
analysing a first portion of the released sample;
collecting a second portion of the released sample;
re-releasing the collected second portion of the sample; and
analysing the re-released portion of the sample.

The sampling tube is typically selected from a plurality of tubes stored in an autosampler.

Preferably, the second portion of the sample released from each

sampling tube is collected either in the sampling tube, or in a separate collecting tube or trap.

Preferably, only a portion of the re-released sample is analysed, a second portion of the re-released sample being re-collected, either in the sampling tube or in a further respective re-collecting tube, each of said respective re-collecting tubes stored either in the same autosampler as the sampling tubes and/or collecting tubes, or in a further autosampler.

It is preferred that the sample released from the sampling tube is buffered, typically by collecting the sample in an intermediate tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the second portion of the released sample.

Alternatively, the second portion of the sample released from the sampling tube, or subsequently collected and re-released, may be buffered, typically by collecting the sample in an intermediate tube or trap, prior to its collection/recollection.

It is particularly preferred that the method is carried out using the analytical apparatus substantially as described hereinbefore.

Embodiments of the present invention will now be described by way of examples only and with reference to the accompanying drawings, of which:

FIGURE 1 is a schematic illustration of an embodiment of apparatus in accordance with the present invention;

FIGURE 2 to 5 are schematic illustrations of respective modes

of operation of the apparatus of Figure 1

FIGURE 6 and 7 are schematic illustrations of respective modes of operation of a second embodiment of apparatus in accordance with the present invention.

Referring to Figure 1, a first embodiment of analytical apparatus in accordance with the present invention is schematically illustrated, the apparatus comprising an autosampler 2, within which a plurality of sorbent tubes are stored (sampling tubes), analysis means 4 and a further sorbent tube (collecting tube) or trap 6. The autosampler may instead be replaced by a single sampling tube.

In a first mode of operation of the apparatus, illustrated in Figures 2 and 3, sampling tubes are automatically selected, in turn, from those stored in the autosampler. Each tube (L) is first heated to desorb a sample from a sorbent material contained therein and the desorbed sample driven from the tube by a flow of inert gas therethrough.

A first portion of the sample driven from the sampling tube (L) is analysed by the analysing means 4 and a second portion passed through the collecting tube (A), within which it is adsorbed by the sorbent material therein or contained in a trap.

The collecting tube (A) is then heated to desorb the sample from the sorbent material therein and is driven out of the tube by a reversed flow of inert gas.

The entire sample thus released from the collecting tube (A) may then be passed through the sampling tube (L), within which it is re-adsorbed for archiving or subsequent analysis (Figure 2), or only a portion of the sample may be passed through the

sampling tube (L) for re-adsorption, with a second portion of the sample being analysed by the analysing means 4 (Figure 3). In the former case the apparatus preferably comprises means for cooling the sampling tube (L) to facilitate re-adsorption by the sorbent material contained therein. In both cases, the apparatus preferably comprises means for cooling the collecting tube (A) to facilitate adsorption by the sorbent material contained therein.

In a first modification of the above process, illustrated schematically in Figures 4 and 5 , the whole (Figure 4) or a portion (Figure 5) of the sample released from the collecting (A) tube may be passed though a sorbent tube (M) other than the sampling tube (re-collecting tube), with which the sampling tube (L) is replaced in the autosampler.

The same process may be repeated for each sampling tube (L,N) selected from the autosampler, either using a single collecting tube (A) for collecting the second portion of the sample released from each sampling tube (Figures 4 and 5) or using a respective collecting tube (A,B) for each sample, as illustrated schematically in Figures 6 and 7, with each collecting tube being selected, in turn, from a plurality of collecting tubes stored in a second autosampler. In the former case, the apparatus preferably comprises means for cooling the collecting tube (A) to facilitate adsorption by the sorbent material contained therein.

As a variation on the processes illustrated in Figures 2, 4, and 6, the entire sample driven from each sampling tube may be collected in a collecting tube, a first portion of the sample subsequently released from the collecting tube being analysed by the analysing means 4, with a second portion of the sample being re-collected in the sampling tube or a re-collection tube. The apparatus preferably comprises means for cooling the

collecting tube to facilitate adsorption by the sorbent material contained therein.

It would also remain in accordance with the present invention, in those embodiments in respect of which the use of only a single collecting tube has been described, for the collecting tube to be substituted with a trap.

The various apparatus thus described provide an efficient and labour saving means for the thermal desorption of a plurality of samples contained in respective sampling tubes, wherein a portion of the desorbed sample is to be collected for subsequent analysis.

CLAIMS

1. An analytical apparatus for automatically carrying out a plurality of analytical steps, which apparatus includes:
a releasing device for releasing a sample from a sampling tube;
a device for analysing a first portion of the released sample;
a collecting device for collecting a second portion of the released sample;
a device for re-releasing the collected said second portion of the sample; and
a device for analysing the re-released portion of the sample.

2. An analytical apparatus according to claim 1, which is arranged to select the sampling tube from a plurality of tubes stored in an autosampler.

3. An analytical apparatus according to claim 1 or 2, which is arranged to provide a comparison of the results from each of the two analysis stages.

4. An analytical apparatus according to any preceding claim, wherein the second portion of the sample released from each sampling tube is collected either in the sampling tube itself or in a separate collecting tube or trap.

5. An analytical apparatus according to claim 4, wherein a single collecting tube or trap is used to collect, in turn, the second portion of the sample released from each of a plurality of sampling tubes.

6. An analytical apparatus according to claim 4, wherein respective collecting tubes are used to collect the second portion of each of the released samples.

7. An analytical apparatus according to claim 6, wherein each of said respective collecting tubes are selected automatically from a plurality of tubes stored either in the same autosampler as the sampling tubes or in a further autosampler.

8. An analytical apparatus according to any preceding claim, wherein a portion of the re-released sample is analysed, a second portion of the re-released sample being re-collected, either in the sampling tube or in a further respective re-collecting tube, each of said respective re-collecting tubes being selected automatically from a plurality of tubes stored either in the same autosampler as the sampling tubes and/or collecting tubes or in a further autosampler.

9. An analytical apparatus according to any preceding claim, arranged such that the sample released from the sampling tube is buffered, by collecting the sample in an intermediate tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the second portion of the released sample.

10. An analytical apparatus according to any of claims 1 to 8, wherein the second portion of the sample released from the sampling tube, or subsequently collected and re-released, are buffered, by collecting the sample in an intermediate tube or trap, prior to its collection/re-collection.

11. An analytical apparatus comprising means for

automatically carrying out, for each of a plurality of sampling tubes stored in an autosampler, the steps of: selecting a sampling tube from said plurality of tubes; releasing a sample from the sampling tube; analysing a first portion of the released sample; collecting a second portion of the released sample, either in the sampling tube or in a collecting tube selected from said plurality of tubes, with which said first tube is replaced in the autosampler.

12. An apparatus according to claim 11, which further comprises means for automatically carrying out the further steps of releasing the sample collected in either the sampling tube or the collecting tube and analysing the released sample.

13. An apparatus according to claim 11 or 12, which is arranged to analyse only a first portion of the sample released by said collecting means, a second portion of the released sample being re-collected, either in the sampling tube, the collecting tube or in a re-collecting tube selected from said plurality of tubes, with which either the sampling tube or the collecting tube is replaced in the autosampler.

14. An apparatus according to any of claims 11 to 13, wherein for each of the plurality of sampling tubes, the sample released from the sampling tube is buffered, by collecting the sample in a tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the second portion of the released sample.

15. An apparatus according to any of claims 11 to 13, wherein the second portion of the sample released from

the sampling tube or by the collecting means may be buffered, by collecting the sample in a tube or trap, prior to its collection/re-collection.

16. An automated analytical method which includes:
releasing a sample from a sample tube;
analysing a first portion of the released sample;
collecting a second portion of the released sample;
re-releasing the collected second portion of the sample; and
analysing the re-released portion of the sample.

17. A method according to claim 16, wherein the sampling tube is selected from a plurality of tubes stored in an autosampler.

18. A method according to claim 16 or 17, wherein the second portion of the sample released from each sampling tube is collected either in the sampling tube, or in a separate collecting tube or trap.

19. A method according to any of claims 16 to 18, wherein a portion of the re-released sample is analysed, a second portion of the re-released sample being re-collected, either in the sampling tube or in a further respective re-collecting tube, each of said respective re-collecting tubes stored either in the same autosampler as the sampling tubes and/or collecting tubes, or in a further autosampler.

20. A method according to any of claims 16 to 19, wherein the sample released from the sampling tube is buffered, typically by collecting the sample in an intermediate tube or trap, prior to the steps of analysing the first portion of the released sample and collecting the

second portion of the released sample.

21. A method according to any of claims 16 to 19, wherein the second portion of the sample released from the sampling tube, or subsequently collected and re-released, may be buffered, typically by collecting the sample in an intermediate tube or trap, prior to its collection/recollection.

INTERNATIONAL SEARCH REPORT

International Application No
GB 03/05242

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01N1/00 G01N1/34

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 409 968 B1 (TAKAHASHI KATSUAKI) 25 June 2002 (2002-06-25) the whole document	1-21
X	US 2002/157483 A1 (LIN MIN-NAN ET AL) 31 October 2002 (2002-10-31) abstract page 2, paragraphs '0019! and '0022! page 4, paragraph '0034!	1-21
X	EP 0 459 677 A (THERMEDICS INC) 4 December 1991 (1991-12-04) abstract; figure 1 column 2, line 14 -column 4, line 2	1-21

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
10 March 2004	18/03/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Bockstahl, F

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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